SYNTHESIS AND CHARACTERIZATION OF GLASS PARTICULATE – EPOXY COMPOSITE FOR STRUCTURAL APPLICATION

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIRMENT FOR THE DEGREE OF

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In
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By

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CERTIFICATE

This is to certify that the thesis entitled "**SYNTHESIS AND CHARACTERIZATION OF GLASS PARTICULATE–EPOXY COMPOSITE FOR STRUCTURAL APPLICATION**" Submitted by **MR. ADITYA NARAYAN MAHARANA** in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance. To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

Date: 11.05.2015
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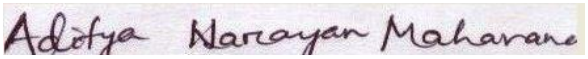

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ABSTRACT:

The objective of this present project work is to prepare and study of the mechanical properties of glass particulate- epoxy composites. A vast amount of research was carried out involving different morphologies of the glass particulates, for e.g. spherical, flakes, rounded etc., for synthesizing epoxy-glass composites. But in this thesis, crushed laboratory waste glasses with acicular morphology were used as a reinforcement to prepare polymer matrix composite material. Five different compositions of the glass particulate, viz., 0, 5, 10, 15 and 20 vol% have been used in the present work. Detailed characterizations like density measurement, X-ray analysis was done for these composite materials, subsequently these polymer matrix composites with different content of glass particulate were subjected to various types of mechanical testing. It was found that tensile properties were increasing from 0 to 5 vol% of reinforcement content, after that a decreasing trend was observed. But other properties like flexural, impact, compression properties were decreasing as compared to the epoxy resin. This work also described about the comparison of the mechanical properties of composites before and after post curing, carried out at 100^{o}C for 15 minutes.

Keywords: Glass-particulate, Polymer matrix Composites, epoxy resin, hardener, post curing

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INTRODUCTION

1.1 Project Topic

Synthesis and Characterization of Glass particulate-Epoxy Composite for Structural Application.

1.2 Aims of the Project

- To utilize laboratory glass wastes by the manufacturing of composites which may be applicable for various structural application
- To reduce the cost of epoxy resin composites by adding more and more glass powder into it so that the strength of the composites is maintained accordingly.
- To study different mechanical properties of glass particulate filled polymer matrix composites.

1.3 Introduction to Composites

As per the ASTM D 3878- 95c, the composites can be defined as "the substance consisting of two or more material, insoluble in one another, which are combined to form a useful engineering material, possessing certain properties not possess by the constituents".

In other words the composites can be defined as the materials which are made from two or more than two constituent materials having different physical and chemical properties from each other individually. When they combine to give a new material the new material's property will be different from the parent two material's by any aspects.

Another definition says that a composite is consisting of a matrix and a reinforcement which are physically different phases whose combination provides a synergistic effect.

Advantages of Composites

The new material formed by combination of two or more constituents is having better properties as compared to the parent materials. The properties include: stronger, lighter, less expensive as compared to the traditional materials. One of the biggest advantages of the modern composite is that they are light as well as strong. The composite provides design flexibility for structural and any other applications.

The main advantages of composites are given below.

I. Flexibility
II. Simplicity
III. Efficiency
IV. High specific properties (High strength to weight ratio)
V. Longevity
VI. Better fatigue and creep properties than their monolithic part etc.

Examples of composites

i. Natural composites

These composites exist in both animals and plants. For example of a natural composite is wood. It is made from long cellulose fibers which are held together by lignin. Cellulose and lignin together form a much stronger substance. But individually they are weak.

Another example of natural composite is bone which is present inside our body. It is made from a material known as "Hydroxyapatite" which is a brittle material and "Collagen" which is a soft and a flexible material. When these two materials combine then strength and properties comes which are needed to support the body

ii. Modern composites

The first modern composite material manufactured was fiberglass. It is used now-a –days in marine applications like in boat hulls, in sports equipment, building panels and in many automobile applications in car bodies. Now- a –days various composites are manufactured and they are applied for various applications. Examples of modern composites are carbon fiber composites, FRP, GFRP etc.

1.4 Classification of Composite

Composite materials are classified as following two distinct parts:

1. **First level of classification:** This is generally done with respect to the matrix constituent present in the composite. The major composites include- i) Metal Matrix Composites MMC) ii) Ceramic Matrix Composites (CMC) iii) Polymer Matrix Composites (CMC). This PMC can be said as Organic Matrix Composites (OMC). Apart from that Carbon matrix Composites are commonly called as Carbon-Carbon composites. The following figure shows the classification of composites by the matrix constituents' point of view.

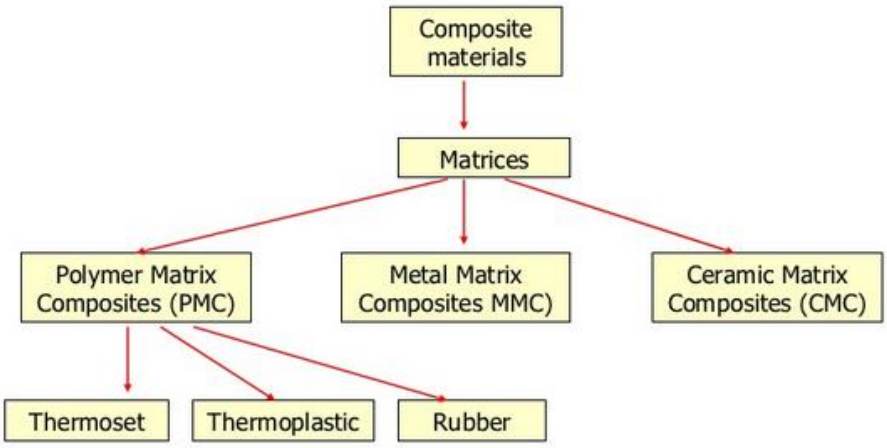


Fig-1.1 Classification of Composites with respect to matrix constituents

2. **Second level of classification:** This level of classification is based on the reinforcement which is present in the composite. These are of three types-**fibre reinforced composites, laminar/structural composites** and **particulate/particle reinforced composites.** Again each category has been divided into sub categories as given in following figure.

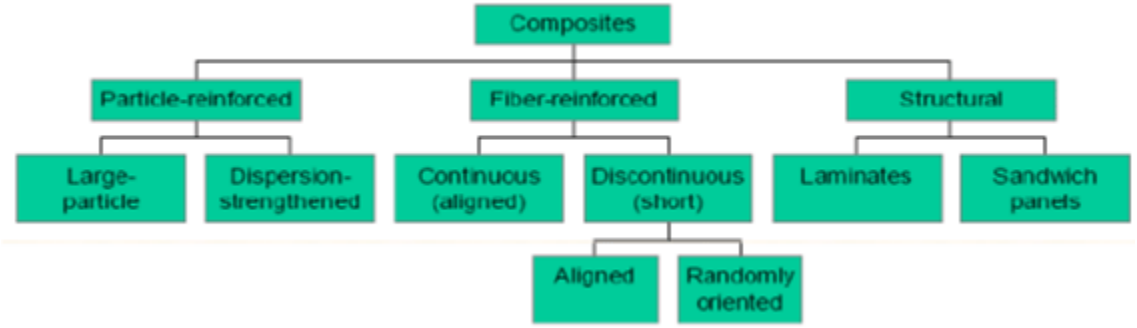


Fig-1.2 classification of composites with respect to reinforcement constituents

But here the matrix constituents' composites are briefly discussed. As we stated above there are three kinds of matrix composites.

1) Metal Matrix Composites: They are composed of a metal matrix and a reinforcement which has excellent mechanical properties, and can be classified whether the reinforcement is continuous or discontinuous. The main matrix materials for MMCs are aluminum and its alloys. Also magnesium and titanium are also used, and for several applications copper, zinc or lead matrixes are used. Not only the single metals but also the systems of metals like, Cu-Zn-Al, W-Co-Cr, Fe-Ni-Zn etc. can be used for the matrix elements. The reinforcement may be either of ceramic materials or any polymer materials. If the ceramic material acts as reinforcement then it is called as *cermet.* There are different advantageous properties of MMC as compared to the other two composites and to the metal counterpart.

- Specific strength is high
- Increased specific stiffness
- Improved wear resistance
- Lower density
- Good corrosion and erosion resistance

- Good electrical and thermal conductivities etc.

But there are some disadvantages of MMC like ease of fabrication is tough as compared to PMC and high energy is required to produce MMC which are the drawbacks of MMC than PMC.

2) Ceramic Matrix Composites (CMC): They are also known as **Ceramic fiber reinforced ceramic** as it contains both ceramic materials in matrix and in reinforcement. Actually the reinforcement in CMC is the ceramic fibers like alumina fibers, silica fibers, silicon carbide fibres, carbon fibres, mullite fibres etc. Also the matrix materials are the same that is mullite, carbon, alumina etc. There are various manufacturing processes applied for producing CMCs like pyrolysis, sintering, CVD, electrophoretic deposition etc. There are various advantageous properties of CMC like-
   - High fracture toughness
   - High tensile strength
   - High bending strength
   - Excellent insulating properties
   - High TSR(Thermal Shock Resistance)
   - High refractoriness
   - High corrosion resistance etc.

But there are few drawbacks of CMCs like the oxidation resistance is very poor(from room temp. to 1000^0C),low flexural strength, brittleness etc. which make them limited use for various applications.

3) Polymer Matrix Composites(PMC): In this type of composites there are three types of polymers which are classified as i) Thermosetting polymers ii) Thermoplastic polymer iii) Rubbery polymers come to the application. Individually or the combination of two or three can be used as the matrix material. These along with ceramic fillers used for various applications. The most important material in these polymers is the resin which is also called as polymer resin. The polymer resin is selected due to some advantages like-

- Mechanical strength comparable to the fillers
- Low shrinkage
- High stiffness with filler materials
- Fatigue resistance
- Heat resistance

- Chemical and moisture resistance etc.
- Good electrical insulating properties

Now-a-days PMCs are used more commercially because it has some advantages over the other two composites type that they have the manufacturing process is economically far better than the other two. Also they have the comparable strengths like the CMCs and MMCs.

Applications of Composites

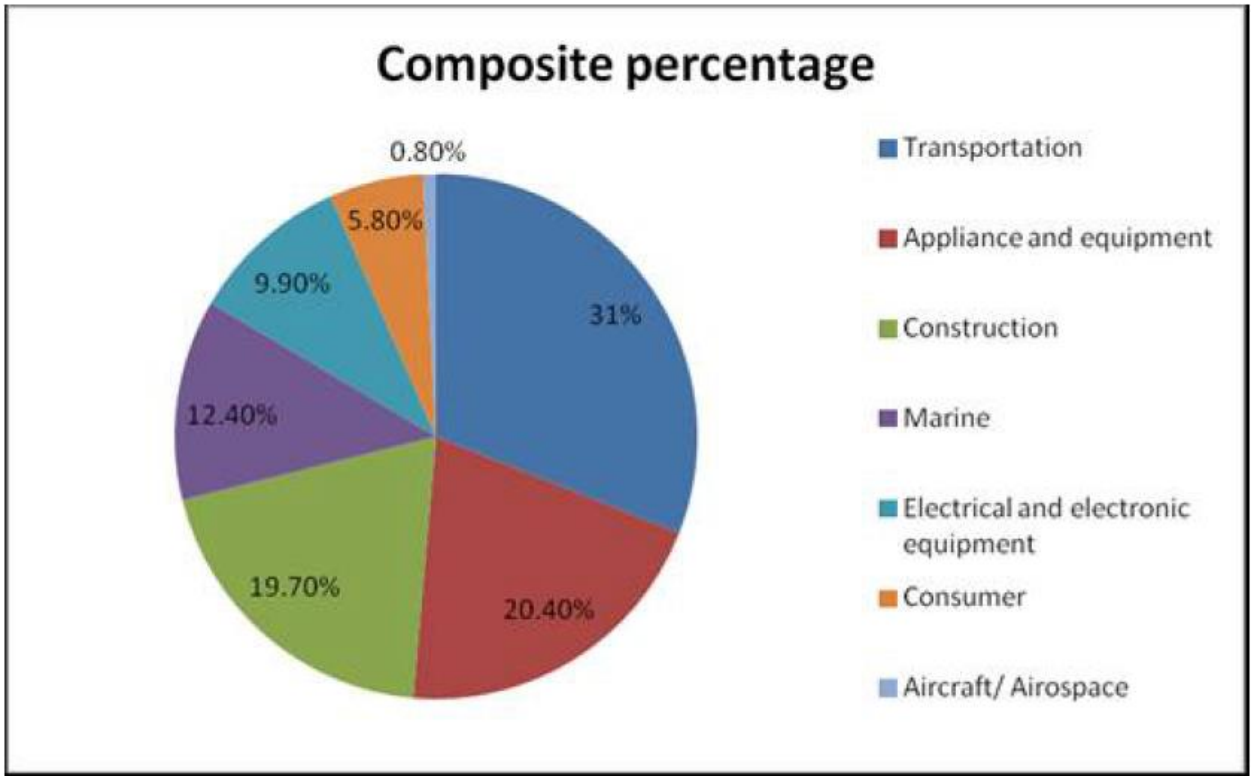


Fig-1.3: Applications of composite in different industries

(Source: SPI composite institute report 2009)

1.5 Epoxy Resin

The name epoxy comes from the word "Epoxide". Epoxide is a functional group which is formed by the reaction of ozone with the unsaturated hydrocarbons. In other words, epoxies are the polyethers having monomer units have an ether type of structures with oxygen bonds, R—O—R whereas the building blocks and chemical reactions involved in producing and crosslinking of unsaturated polyesters are similar for different polyester types, the situation is rather complex

with epoxies. The epoxide group contains one oxygen atoms and two or more carbon atoms in a ring type of structure as shown in follow.

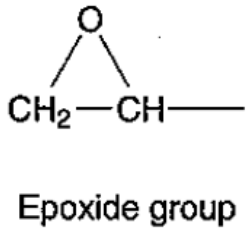


Fig- 1.4: Structure of epoxide group

1.6 Fillers

Fillers are mixed to epoxy resins to improve certain physical properties like adhesion, stiffness, strength etc., also to improve versatility and to reduce the cost of materials. According to the application point of view we add fillers by some percentages to the epoxy resin. For example silica is added to epoxy resin to increase its density and to stiffness which will be used in filleting materials and in wall putties. Graphite powder is also added to epoxy resin to make low friction coating. Carbon fibers are added to epoxy to increase the chemical resistance of epoxy resin.
In the present study the glass powders which is discussed is the waste glass which come from the laboratory wastes.

Hardener

These are known as the curative agents for the epoxy resin. Generally the uncured epoxy resins have low mechanical, chemical and heat and corrosion resistance properties. When the hardener is added to the epoxy then good properties are obtained. This is because of the reaction of the hardener with the epoxy to form a new cross-linked suitable three- dimensional structure which has thermosetting behavior. The above process is generally called as curing. This curing process is an exothermic process. The hydrogen atom present in the hardener generally reacts with the epoxide groups of the epoxy resin to form this thermosetting crosslinking polymer. Some examples of commercial hardeners are HY951, HY840, HY5200, HY 956 etc.

Chapter 2

LITERATURE REVIEW

An exhaustive amount of work was done in this field; few of these are delineated below:

- **R. Taurino and *et.al*[1]** worked on the new composite materials based in the glass waste and they found the advantage in using coarse glass particles in bi-layer composite was the energy saving in the glass manufacturing step, since no much more processing was required. The addition of waste glass fillers to the resin matrix resulted in a reduction of flexural strength, but an increase of elastic modulus and dynamic stiffness were obtained in the bi-layer composite.

- **Metin Sayer[2]** worked on the Elastic properties and buckling load evaluation of ceramic particles filled glass/epoxy composites and he found that the elasticity moduli and load carrying capability of composites were significantly influenced by particle weight fractions, different particle sizes and different ceramic particles.

  In general, the addition of ceramic particles like glass particulates to composites increased the elasticity moduli and load carrying capability of composites. Accordingly, all composites with 10 weight% filler had maximum elasticity moduli values and the best ability to resist buckling load.

- **Eric Minford *et.al* [3]** worked on method of making hybrid composite structures of fibre reinforced glass and resin matrices and found that the fibre reinforcement in the resin matrix composite may comprise any fibre that exhibited a tensile strength greater than about 10×10^3 psi, a tensile modulus greater than about 10×10^6 psi, thermal stability at temperatures up to about 700° C. and is wettable by the matrix material.

- **Siriporn Damrongsakkul *et.al* [4]** worked on thermo mechanical and rheological behaviours of waste glass particulate filled polyester resins composites and found that the addition of glass particulate waste obtains from surfboard manufacturing industry was able to reduce the heat of fusion of polyester resins composite because of the decrease in the amount of polyester resins .

Furthermore, the tensile and flexural properties of the composites were increased with increasing the glass particulate contents. The results from rheological studies were able to provide the apparent flow activation energy, which could reveal that the addition of glass fibre decreased the fluidity of the molten composite materials .

- **Venceslav Vassilev and *et. al.* [5]** worked on Composites Containing Waste Materials and they found that the composite materials developed on the basis of unsaturated polyester resins showed relatively good strength characteristics and could find application in the machine-building industry for the production of housings and other parts, replacing other materials with similar parameters, but of higher cost.

  They also found that the materials based on epoxy resins are better electro insulators and could be successfully used for electrical insulation compounds with application in electrical constructions, radio-electronics circuitry and other types of electrical equipment and appliances**.**

- **P. Valasek and *et.al*[6]** worked on polymeric composite based on glass powder – usage possibilities In agro complex and they found that the interaction of the glass powder and the epoxy resin represents an interesting way of the material recyclation which is sensitive to the environment.They also found the characteristics properties of the composite system based on the glass powder can be summarized as follows:

  - increased hardness, abrasive wear resistance at preserving cohesive and adhesive qualities
  - decreased impact strength
  - acceptable costs, available and easy application.

- **M. Sanchez-Soto et *al.* [7]** worked on study and mechanical characterization of glass bead filled tri-functional epoxy composites and they found that both the Young's modulus and the resin strength at break were improved by the addition of untreated glass-beads. Also the Young's modulus increased with the volume of glass filler added.

- **J.Z. Liang and *et al.*[8]** researched on the topic of measurement of thermal conductivity of glass-bead-filled polypropylene composites. They came to a conclusion that the heat insulation property of polymeric materials might be improved by filling with inorganic glass particulate phase. They also found that the effective thermal conductivity of glass-bead-filled polypropylene (PP) composites decreased roughly linearly with increase of the volume fraction of the beads.

- **A. A. Ibrahim and *et al.* [9]**worked on flexural properties of glass particles filled polymer composites and he came to the conclusion that the hybrid composite reinforced with 10% glass particles presented the best overall flexural properties. It had the highest ultimate flexural strength as well as an excellent stiffness and a strain to failure comparable to that of the polymer matrix material.

- **Kwok Yeung Peter Wong and *et al.* [10]** researched on measurement of mechanical, electrical and thermal properties of glass powder reinforced epoxy composites and he found that composite with 5 % weight of glass powder post-cured in micro oven had shown comparatively good electrical, mechanical and thermal properties. It could reduce the overall cost of material without much affecting desirable properties of the material.

Chapter 3

EXPERIMENTAL PROCEDURE

Principle Involved:

The addition of glass particulate in epoxy composite, mechanical properties gets enhanced to some extent when compared with the only epoxy. After a certain amount of addition of glass particulates these properties do not get enhanced and in some cases on further addition of fillers the properties get decreased.

Chemical ingredients used:

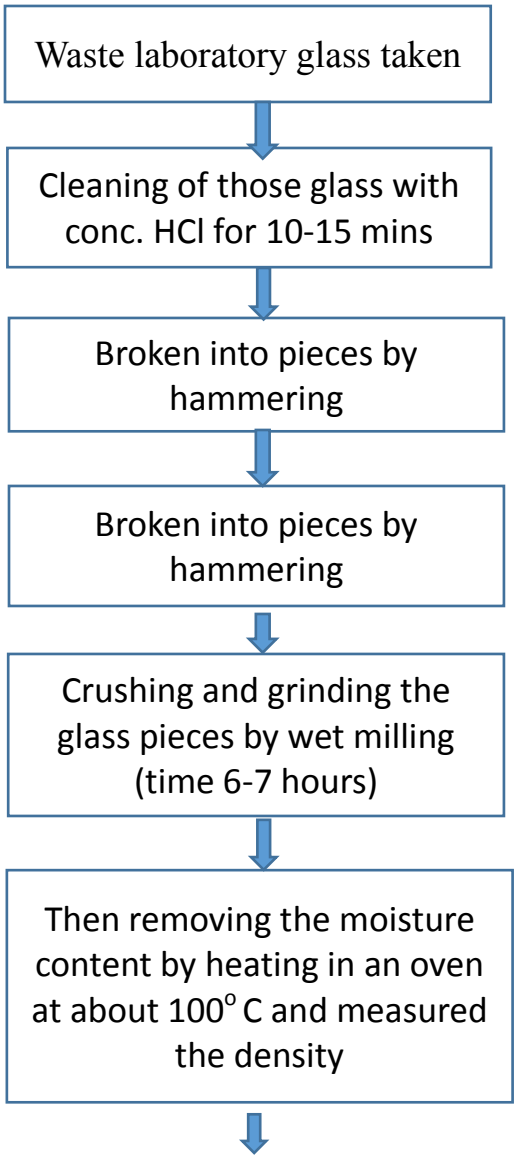
1. Epoxy Resin (AW-106)
2. Hardener (HY-953)
3. Silicon Spray
4. Glass powders
6. Dilute HCl for cleaning of waste glass beaker

Apparatus Used:

1. Container i.e., steel bowl for wet milling
2. Silicon nitride balls i.e. grinding media
3. Weighing machine
4. Wooden board
5. Transparent plastic Sheet
6. Plastic glass (Use and throw)
7. Nails
8. Wooden bit
9. PVC pipes
10. Hammer
11. Hack saw
12. File
13. Amery papers for polishing
14. UTM
15. Vickers Hardness Testing Machine
16. Impact Testing Machine
17. Malvern Particle size analyser
18. DSC/TG machine
19. XRD machine

The procedure for making the composites and their characterization is given briefly in a following flow chart.

3.1 Process Flow Chart for Manufacturing of Composite



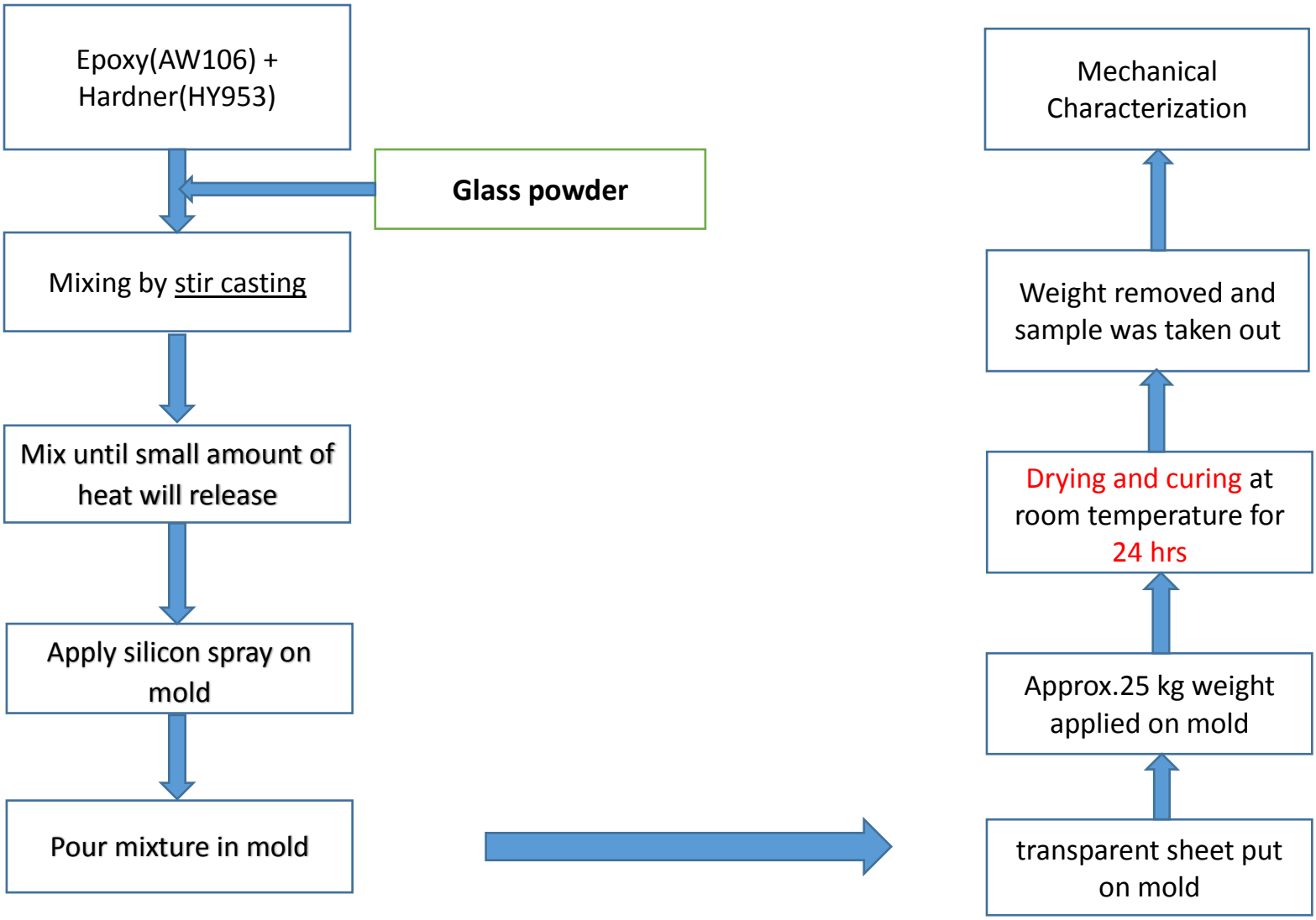


Fig-3.1 Process Flow Chart for Manufacturing of Composite

3.2 Detailed Procedures for making the composite

3.2.1 Determination of particle size of glass powder:

It was done by Malvern Particle size analyzer. First the glass powder which was crushed and ground into very fine particle were dispersed in water. Then this was ultrasonicated for 15-20 minutes. Then the particle size was determined. The average particle size of the glass powder was found to be 1.296 micron.

3.2.2 Measurement of Density of glass powder:

It was done by pycnometric method. In this method the glass powder (sample) having 1.296 micron was poured into a 10 ml pycnometric bottle. One–third of the volume of the glass powder was taken in this bottle. Then half volume of the bottle was filled and then entire bottle was kept in vacuum atmosphere so that extra bubble entrapment would come out. This procedure was followed for 2 hours. After that weights were taken

Let the weight of empty bottle= W_1=25.0608gm

Let the weight of sample+ bottle = W_2 =30.5035gm

Let the weight of bottle + sample + water = W_3 = 51.7911gm

Let the weight of bottle + water = W_4 = 48.8034gm

Now the actual weight of the sample= (W_2-W_1) gm= 5.4427 gm

Weight of Liquid(in this case water) = (W_4-W_1) gm=23.7426 gm

Weight of water other than sample volume= (W_3-W_2) gm= 21.2876 gm

Now the specific gravity of sample is given by

$$(W_2-W_1)/ (W_4-W_1)- (W_3-W_2)$$

$$=2.2169$$

So the density of the sample = specific gravity of the sample × Density of water at R.T

$$= 2.2169 \times 0.99 \text{ g/cm}^3$$

$$= 2.22 \text{ g/cm}^3$$

The density of the waste glass powder was found to be 2.22 g/cm^3.

3.2.3 Mold preparation and calculation of required amount of filler, epoxy resin and hardener

i) The mold was prepared which would be essential for the preparation of composite. The molds were rectangular in size having dimensions= 150mm×65mm×7mm

The mold was prepared by the help of the wooden bits which were placed on a wooden board.

Figure 3.2: mold prepared for the manufacture of composite

Before mold preparation a transparent plastic sheet was introduced on the top layer of the wooden board so that the epoxy would not stick to this. After the mold preparation required amount of glass powder, epoxy resin and hardener which would be required were calculated.

Calculation of required glass powder, epoxy resin and hardener:

a) For tensile and flexural testing-

Density of waste glass powder (obtained from pycnometry) = 2.21gm/cc

Density of epoxy resin (**AW 106**) = 1.17gm/cc

Density of hardener (**HY953**) = 0.953 gm/cc

Now the volume of the mold= 150mm×65mm×7mm = 68250mm^3

The ratio of epoxy to hardener added = 10:8

So the percentage of hardener = (8/18)×100 = 44.44%

The percentage of epoxy = (10/18)×100 = 55.55%

Now the density of matrix was given by,

ρ_{matrix} = (44.44/100)×0.953 + (55.55/100)×1.17

= 0.41932 + 0.6435

= 1.06283 gm/cc

Now a) for 0 vol% glass powder used as filler,

Glass powder required = 0 gm

Mass of matrix = 1.06283 × 68250 × 10^{-3} gm

= 72.5381 gm

Now mass of epoxy required = (10/18)×72.5381 gm = 40.298 gm

Mass of hardener required = (8/18) × 72.5381 gm = 32.23917 gm

b) for 5 vol% glass powder used as filler,

Glass powder required = $2.21 \times 0.05 \times 68250 \times 10^{-3}$ gm = 7.5416 gm

Mass of matrix = $0.95 \times 1.06283 \times 68250 \times 10^{-3}$ gm

= 68.911 gm

Now mass of epoxy required = (10/18)× 68.911 gm = 38.283 gm

Mass of hardener required = (8/18) × 68.911 gm = 30.62711 gm

c) for 10 vol% glass powder used as filler,

Glass powder required = $2.21 \times 0.10 \times 68250 \times 10^{-3}$ gm = 15.0832 gm

Mass of matrix = $0.90 \times 1.06283 \times 68250 \times 10^{-3}$ gm

= 65.284 gm

Now mass of epoxy required = (10/18)× 65.284 gm = 36.26 gm

Mass of hardener required = (8/18) × 65.284 gm = 29.015 gm

d) for 15 vol% glass powder used as filler,

Glass powder required = $2.21 \times 0.15 \times 68250 \times 10^{-3}$ gm = 22.6248 gm

Mass of matrix = $0.85 \times 1.06283 \times 68250 \times 10^{-3}$ gm

= 61.657 gm

Now mass of epoxy required = (10/18)× 61.657 gm = 34.25388 gm

Mass of hardener required = (8/18) × 61.657 gm = 27.40311 gm

e) for 20 vol% glass powder used as filler,

Glass powder required = $2.21 \times 0.20 \times 68250 \times 10^{-3}$ gm = 30.1664 gm

Mass of matrix = $0.80 \times 1.06283 \times 68250 \times 10^{-3}$ gm

= 58.030 gm

Now mass of epoxy required = (10/18)× 58.030 gm = 32.23 gm

Mass of hardener required = (8/18) × 58.030 gm = 25.791 gm

The details of result obtained from calculation have been summarized below in a table.

Table- 3.1: amount of ingredients required to manufacture the composite for tensile and flexural testing

Vol % of glass powder	Weight of glass powder required (in gm)	Weight of epoxy required(in grams)	Weight of hardener(in gm)
0	0	40.298	32.23917
5	7.5416	38.283	30.62711
10	15.0832	36.26	29.015
15	22.6248	34.25388	27.40311
20	30.1664	32.23	25.791

The above table showed only the calculations for the samples which would be tested for tensile and flexural testing. But for the compression and impact testing the calculations would be different because the size of the mold varies.

It is noted that the maximum vol% of glass powder for the manufacturing of composites in this grade of epoxy was 20 vol%. After that the slurry would be more viscous so that it could not be casted.

ii) then the epoxy and hardener were mixed in a use and throw plastic glass. After some time small heat was generated due to the reaction between epoxy and hardener.

iii) Then required amount of glass powder were added to the slurry and again stirred properly.

iv) Then after the slurry was poured into the mold and upon that a transparent sheet was applied to make the surface smooth.

v) After that about 25 kg of load was applied on the body and it was kept in room atmosphere for curing and setting for 24 hours.

Fig-3.3: slurry after casing

Fig-3.4: load was applied for curing and setting

vi) After 24 hours curing the sample was taken out and from that 4 pieces of each having dimensions = 150mm×15mm×7mm were cut. Again from that, length of the four cut pieces was reduced to 75mm. By the way total 8 samples were obtained. 4 samples for tensile and other four samples were for flexural testing.

vii) The four samples which were cut for the tensile testing again they were cut and polished to form a dog- bone shape. the dimensions of this samples were = samples=75mm×10mm×7.5mmwhere gauge length=40mm.The image was given below

Fig-3.5 images of dog bone samples which were used for tensile testing

But for the Flexural testing no dog-bone structure was required. They were just polished by the help of a hand file. The image which was given below shows the flexural specimens.

Fig-3.6 Samples which were used for flexural testing

b) For compression testing-

The calculation for samples for compression testing was quite different from the earlier two samples. It used a PVC pipe which were cut into pieces where the inner diameter of the pipe was 17mm. the length/height was kept about 3-4 mm greater than the diameter so that after polishing the dia/height ratio would nearly same as unity (i,e, l/d =1).

Calculations for compression mold was given below.

Diameter of the mold = 17mm ; Height taken = 21mm,

Volume of mold = $\pi \times \frac{d\hat{}2}{4} \times h$ = 4766.581mm^3

where d=diameter of mold and h= height of mold

i) <u>For 0 vol% glass powder used as filler,</u>

Glass powder required = 0 gm

Weight of matrix = 1×1.06283×4766.581×10^{-3} gm = 5.066gm

Mass of epoxy would be = (10/18)×5.066gm = 2.81gm

Mass of hardener would be= (8/18)× 5.066gm = 2.256gm

ii) <u>For 5 vol% glass powder used as filler,</u>

Glass powder required = 0.05×2.21×4766.581×10^{-3}gm = 0.526 gm

Weight of matrix = 0.95×1.06283×4766.581×10^{-3} gm= 4.812gm

Mass of epoxy would be = (10/18)× 4.812gm = 2.67gm

Mass of hardener would be= (8/18)× 4.812gm = 2.2138gm

iii) <u>For 10 vol% glass powder used as filler,</u>

Glass powder required = 0.10×2.21×4766.581×10^{-3}gm = 1.053 gm

Weight of matrix = 0.90×1.06283×4766.581×10^{-3} gm= 4.559gm

Mass of epoxy would be = (10/18)× 4.559gm = 2.533gm

Mass of hardener would be= (8/18)× 4.559gm = 2.02gm

iv) <u>For 15 vol% glass powder used as filler,</u>

Glass powder required = 0.15×2.21×4766.581×10^{-3}gm = 1.578 gm

Weight of matrix = 0.85×1.06283×4766.581×10^{-3} gm = 4.306gm

Mass of epoxy would be = (10/18)× 4.559gm = 2.392gm

Mass of hardener would be= (8/18)× 4.559gm = 1.913gm

v) For 20 vol% glass powder used as filler,

Glass powder required = $0.20\times2.21\times4766.581\times10^{-3}$gm = 2.2106 gm

Weight of matrix = $0.80\times1.06283\times4766.581\times10^{-3}$ gm = 4.052gm

Mass of epoxy would be = (10/18)× 4.052gm = 2.251gm

Mass of hardener would be= (8/18)× 4.052gm = 1.800gm

The detailed calculations were summarized in the below table-

Table- 3.2: amount of ingredients required to manufacture the composite for compression testing

Vol % of glass powder	Weight of glass powder required (in gm)	Weight of epoxy required(in grams)	Weight of hardener(in gm)
0	0	2.81	2.256
5	0.526	2.67	2.138
10	1.053	2.533	2.02
15	1.578	2.392	1.913
20	2.106	2.251	1.800

viii) After calculating the gradients for compression samples required amount of epoxy and hardener were mixed in a plastic use and throw glass as per the procedure of the earlier cases then glass powder was mixed and stirred thoroughly.

ix) Then the slurry was poured to the mold and then cured for 24 hours

x) after 24 hours the samples were taken out from the mold and polished so that the l/d ratio became one.

Fig-3 .7: Compression samples having dimension=17mm×17mm

The other compression sample images are given below.

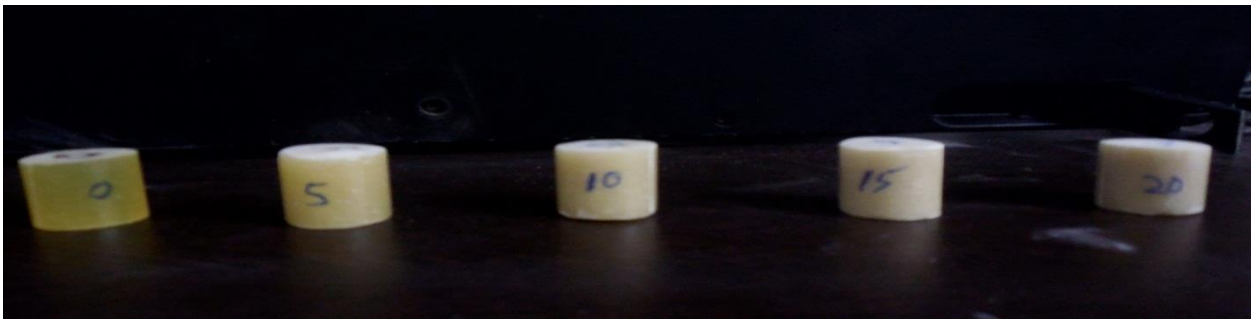


Fig-3 .8: Compression samples (0,5 10,15, 20 vol%)

c) For Impact testing-

In this case the thickness would be different i,e, 5mm. The dimension of the mold would be taken as 65mm×65mm×5mm.

Calculations for impact mold was given below:

Length of the mold = 65mm;

Breadth of the mold = 65mm;

Height of the mold = 5mm;

Now the volume of the mold = 65mm×65mm×5mm = 21125mm^3

i) <u>For 0 vol% glass powder used as filler,</u>

Glass powder required = 0 gm

Weight of matrix = 1×1.06283×21125×10^{-3} gm = 22.452gm

Mass of epoxy would be = (10/18)× 22.452gm = 12.473gm

Mass of hardener would be= (8/18)× 5.066gm = 9.978gm

ii) <u>For 5 vol% glass powder used as filler,</u>

Glass powder required = 0.05×2.21×21125×10^{-3} gm = 2.234gm

Weight of matrix = 0.95×1.06283×21125×10^{-3} gm = 21.329gm

Mass of epoxy would be = (10/18)× 21.329gm = 11.85gm

Mass of hardener would be= (8/18)× 21.329gm = 9.5gm

iii) <u>For 10 vol% glass powder used as filler,</u>

Glass powder required = 0.10×2.21×21125×10^{-3} gm = 4.668gm

Weight of matrix = 0.90×1.06283×21125×10^{-3} gm = 20.207gm

Mass of epoxy would be = (10/18)× 20.207gm = 11.23gm

Mass of hardener would be= (8/18)× 20.207gm = 8.98gm

iv) For 15 vol% glass powder used as filler,

Glass powder required = $0.15\times2.21\times21125\times10^{-3}$ gm = 7.002gm

Weight of matrix = $0.85\times1.06283\times21125\times10^{-3}$ gm = 19.804gm

Mass of epoxy would be = (10/18)× 19.804gm = 11.002gm

Mass of hardener would be= (8/18)× 19.804gm = 8.802gm

v) For 20 vol% glass powder used as filler,

Glass powder required = $0.20\times2.21\times21125\times10^{-3}$ gm = 9.336gm

Weight of matrix = $0.80\times1.06283\times21125\times10^{-3}$ gm = 17.961gm

Mass of epoxy would be = (10/18)× 17.961gm = 9.9783gm

Mass of hardener would be= (8/18)× 17.961gm = 7.98266gm

The detailed calculations were summarized in the below table-

Table- 3.3: amount of ingredients required to manufacture the composite for impact testing

Vol % of glass powder	Weight of glass powder required (in gm)	Weight of epoxy required(in grams)	Weight of hardener(in gm)
0	0	12.473	9.978
5	2.334	11.85	9.5
10	4.668	11.23	8.98
15	7.002	11.002	8.802
20	9.336	9.9783	7.98266

Chapter 4

RESULTS AND DISCUSSIONS

4.1 Characterization of Glass Powder

4.1.1 Density measurement of Glass Powder by Pycnometry-

The density of the glass powder was found to be 2.22gm/cc

4.1.2 Particle size Determination(PSD) of Glass Powder-

The average particle size of the glass powder was found to be 1.296micron. The result has been shown below

Results

			Size (d.nm...	% Intensity:	St Dev (d.n...
Z-Average (d.nm):	1296	Peak 1:	620.7	100.0	91.99
PdI:	0.779	Peak 2:	0.000	0.0	0.000
Intercept:	1.07	Peak 3:	0.000	0.0	0.000
Result quality	Refer to quality report				

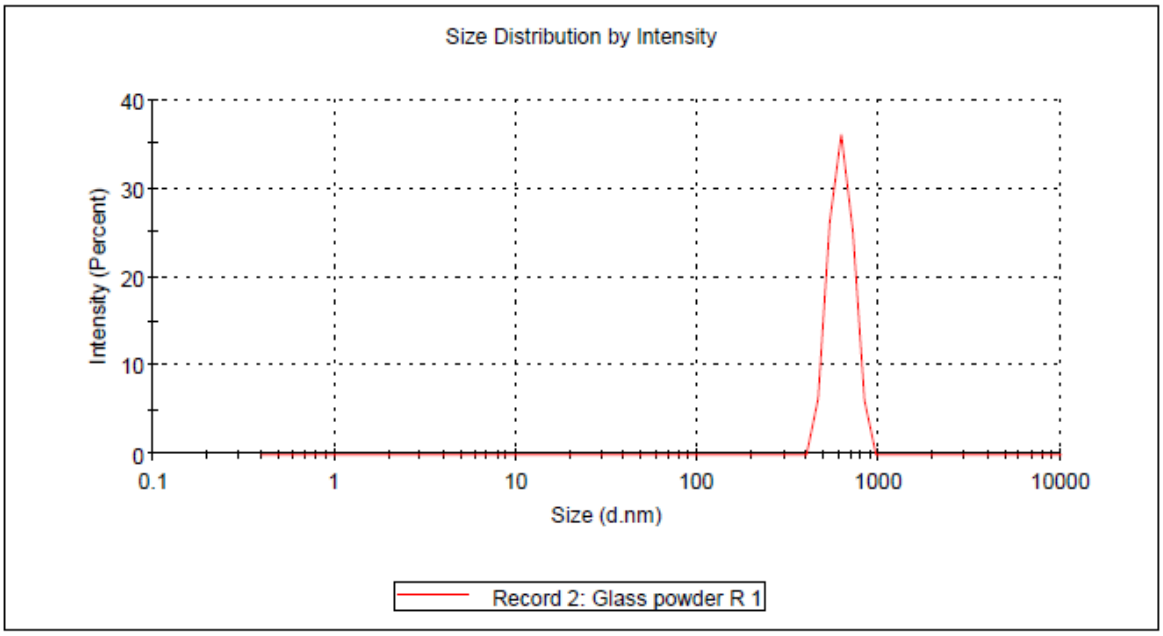


Fig-4.1: the above fig shows the average particle size determination of glass powder

4.1.3 X-Ray Diffraction (XRD) of glass powder-

As we know the glass is an amorphous material, so the XRD analysis showed the broad peak as compared to the crystalline material having sharp peak. The reason of the coming broad peak is that the atoms of the glass powder have short range order and the atoms are randomly oriented. The XRD peak has been given below.

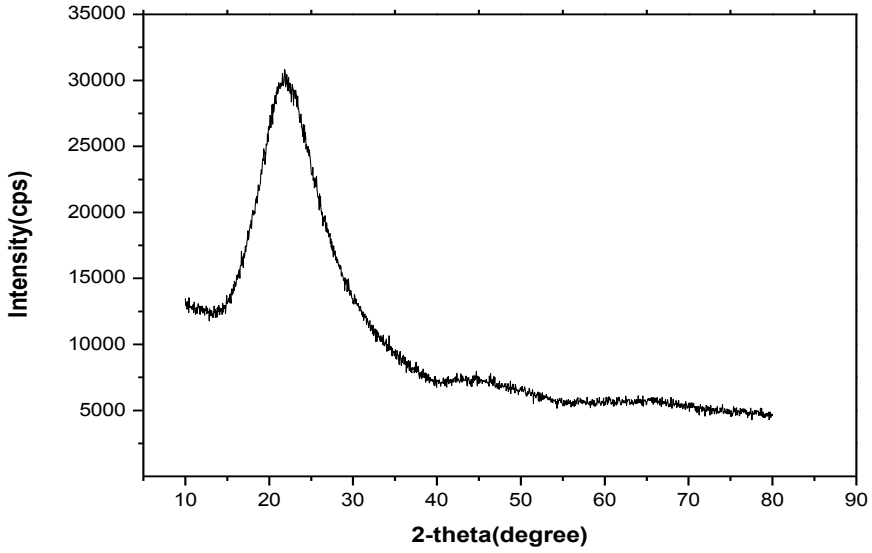
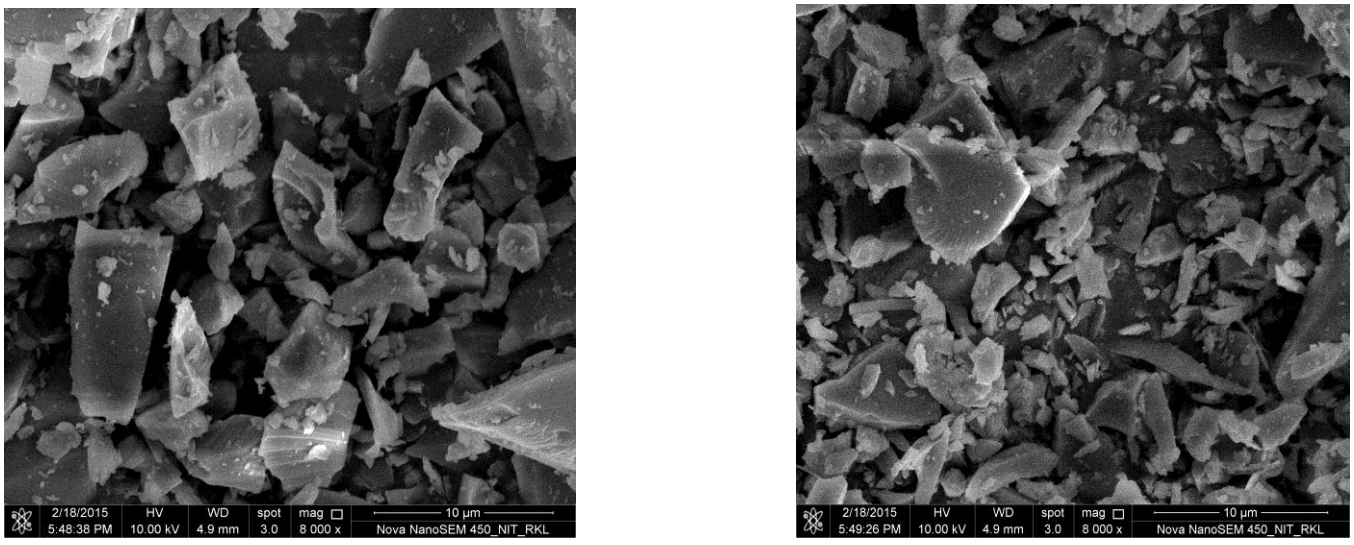


Fig-4.2: XRD peak of the glass powder

4.1.4 Scanning Electron Microscopy (SEM)



(a) **(b)**

Fig-4.3: a) & b) SEM micrographs of glass powder show the glass particles were of different sizes

4.2 Density measurement of Composite

a) By Archimedes Principle: It was done by Archimedes Principle. This principle states that "The weight of the displaced fluid is directly proportional to the volume of the displaced fluid". The formula given below was used to determine the density of an object.

$$\frac{\text{density of object}}{\text{density of fluid}} = \frac{\text{weight}}{\text{weight of displaced fluid}}$$

b) By Rule of Mixture:

i) For 0 vol% glass powder epoxy composite

$\rho_{composite} = \rho_{matrix} V_{matrix} + \rho_{particulate} V_{particulate}$

$= \rho_{epoxy} V_{epoxy} + \rho_{glass\ powder} V_{glass\ powder}$

where ρ, V are the density and volume fraction of each phase

So $\rho_{composite} = (1.072 \times 1) + 0$

$= 1.072$ gm/cc

iI) For 5 vol% glass powder epoxy composite

$\rho_{composite} = \rho_{matrix} V_{matrix} + \rho_{particulate} V_{particulate}$

$= \rho_{epoxy} V_{epoxy} + \rho_{glass\ powder} V_{glass\ powder}$

$= (1.072 \times 0.95) + (2.21 \times 0.05)$

$= 1.1289$ gm/cc

iii) For 10 vol% glass powder epoxy composite

$\rho_{composite} = \rho_{matrix} V_{matrix} + \rho_{particulate} V_{particulate}$

$= \rho_{epoxy} V_{epoxy} + \rho_{glass\ powder} V_{glass\ powder}$

$= (1.072 \times 0.90) + (2.21 \times 0.10)$

$= 1.1858$ gm/cc

iv) For 15 vol% glass powder epoxy composite

$\rho_{composite} = \rho_{matrix} V_{matrix} + \rho_{particulate} V_{particulate}$

$= \rho_{epoxy} V_{epoxy} + \rho_{glass\ powder} V_{glass\ powder}$

$= (1.072 \times 0.85) + (2.21 \times 0.15)$

$= 1.2427$ gm/cc

v) For 20 vol% glass powder epoxy composite

$\rho_{composite} = \rho_{matrix} V_{matrix} + \rho_{particulate} V_{particulate}$

$= \rho_{epoxy} V_{epoxy} + \rho_{glass\ powder} V_{glass\ powder}$

$= (1.072 \times 0.80) + (2.21 \times 0.20)$

$= 1.2996$ gm/cc

The details of densities for both obtained by Archimedes principle and by mixture rule are given below in a separate table.

Table-4.1: Density of composite by Archimedes principle and mixture rule

Vol% of glass powder used as filler in composite	Density,(gm/cc) (By Mixture Rule)	Density,(gm/cc) (By Arcmedis Principle)
0	1.072	1.097
5	1.1289	1.141
10	1.1858	1.189
15	1.2427	1.250
20	1.2996	1.285

The below graph shows the varaitaion of the densities with respesct to vol% of glass powder

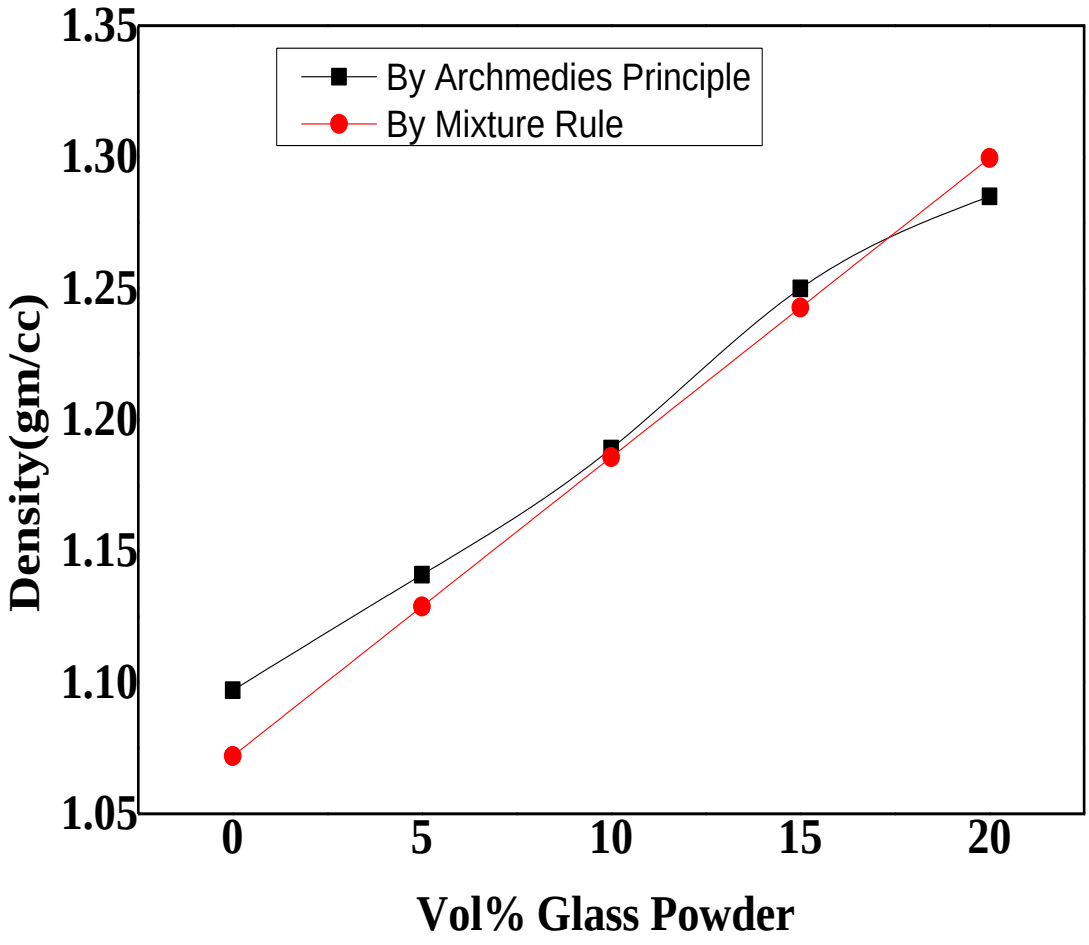
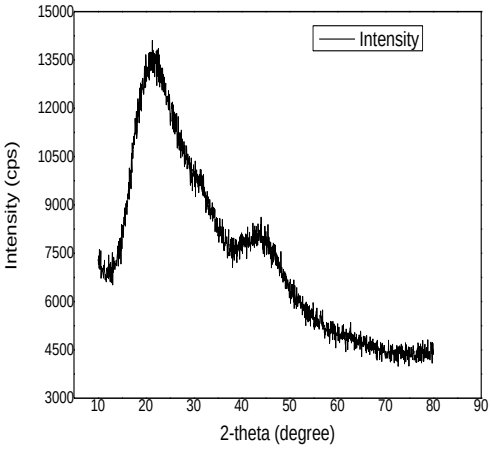


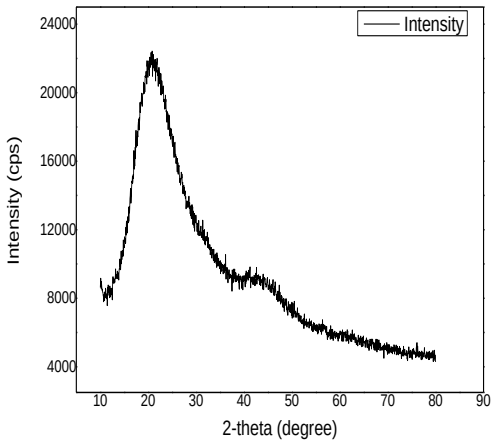
Fig-4.4 variation of density with respect to vol% glass powder

4.3 Mechanical Characterization of Composite

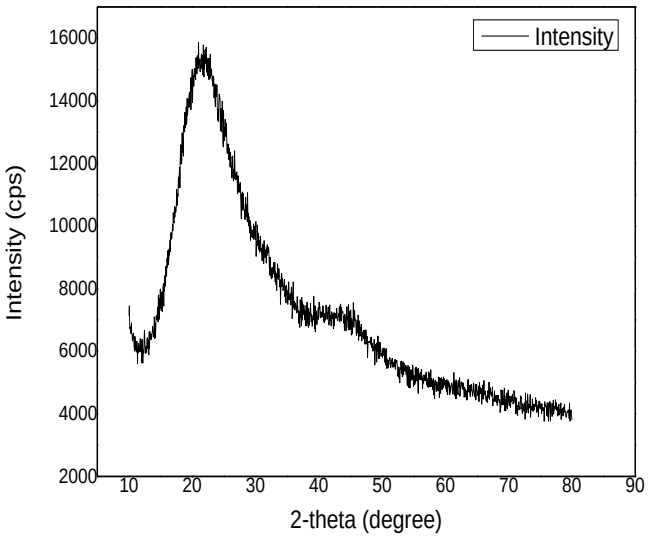
4.3.1 X-Ray Diffraction (XRD)



a) **Fig- 4.5(a)XRD of pure epoxy AW106**



b) fig4.5(b) XRD of 10 vol% GP-epoxy composite

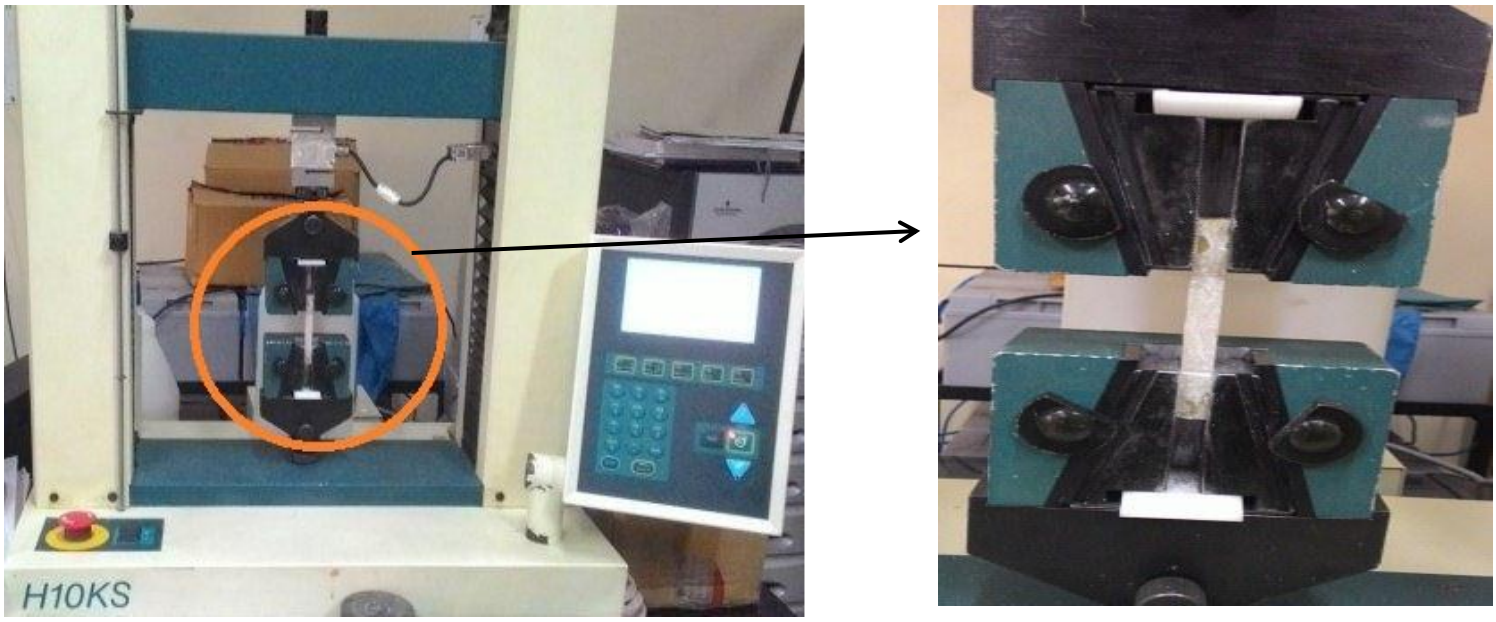


c) fig- 4.5(c)XRD of 20 vol% GP-epoxy composite

4.3.2 Tensile Test

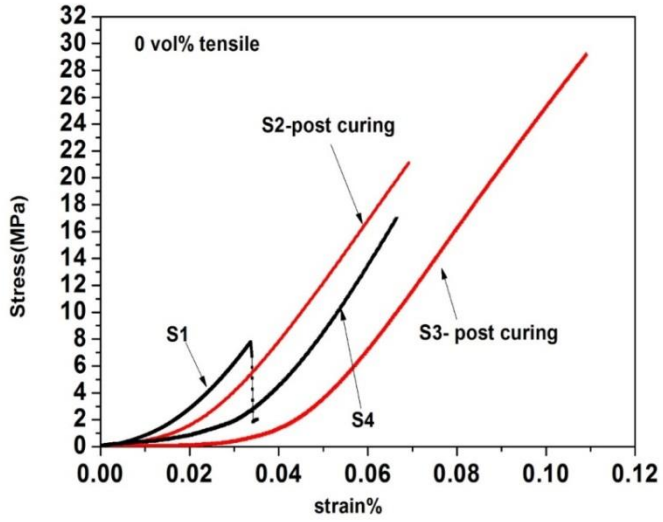
Tensile strength is the optimum stress that can be withstood by a material when this material is stretched before it comes to break. In tensile testing the neck formation occurs and after substantial deformation it comes to break. The tensile test was generally performed by UTM machine (HK10S UTM machine). Mathematically the tensile test is defined as

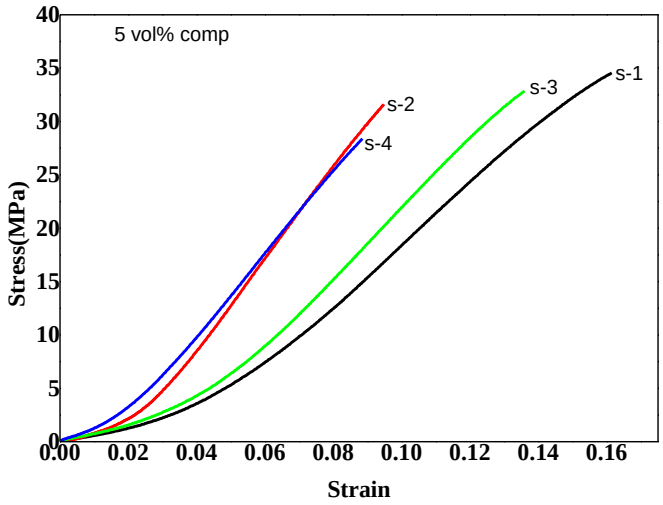
Tensile stress = Load/ initial area of the specimen

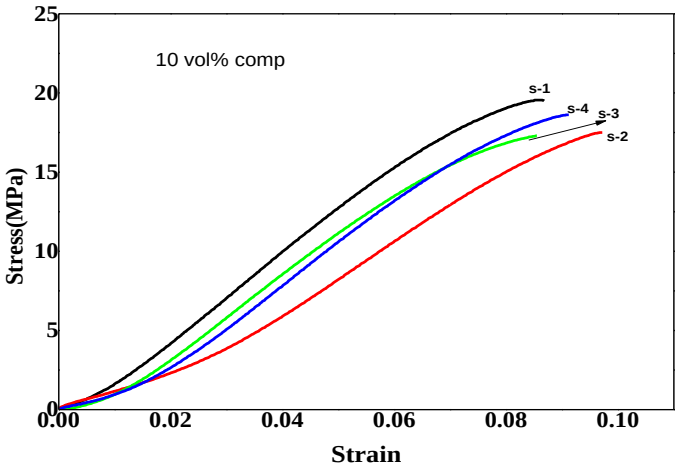


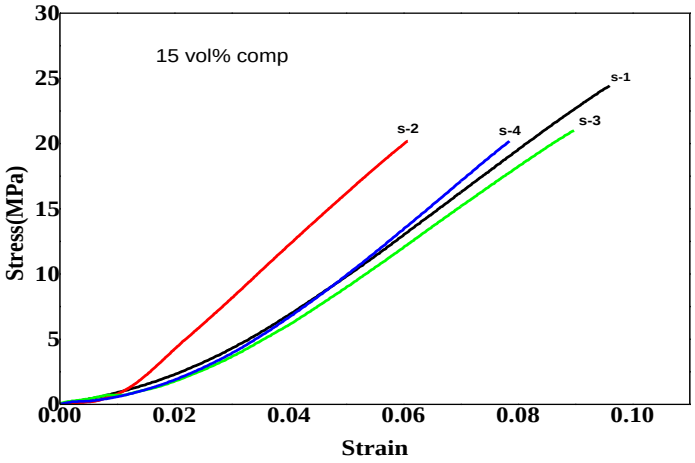
(a) (b)

Fig-4.6 (a) Universal Testing Machine in which the sample is placed;(b) the enlarged portion









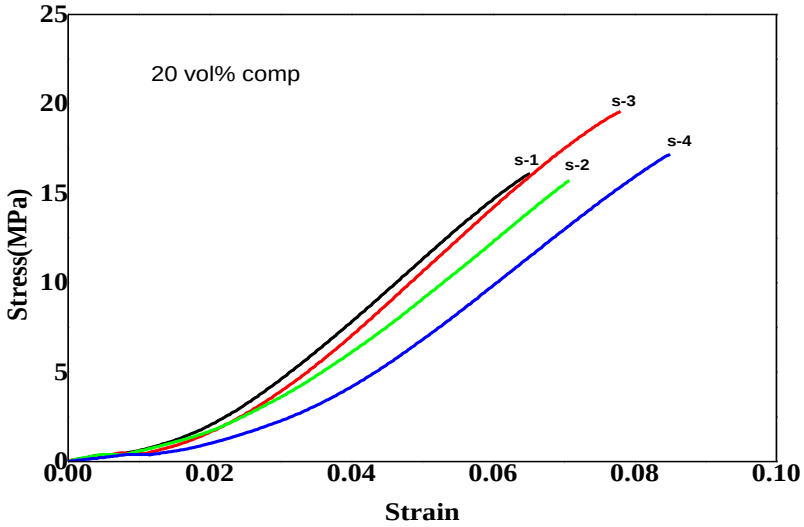


Fig-4.7 Stress vs strain graphs for different vol% of GP epoxy composite(each having 4 samples

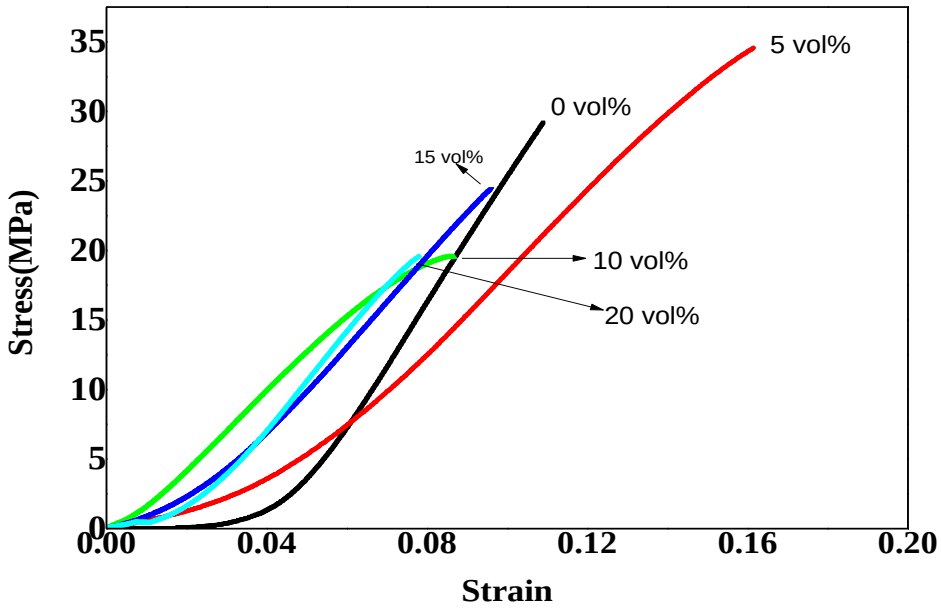


Fig-4.8 Composite graph for all vol% glass powder-epoxy composites

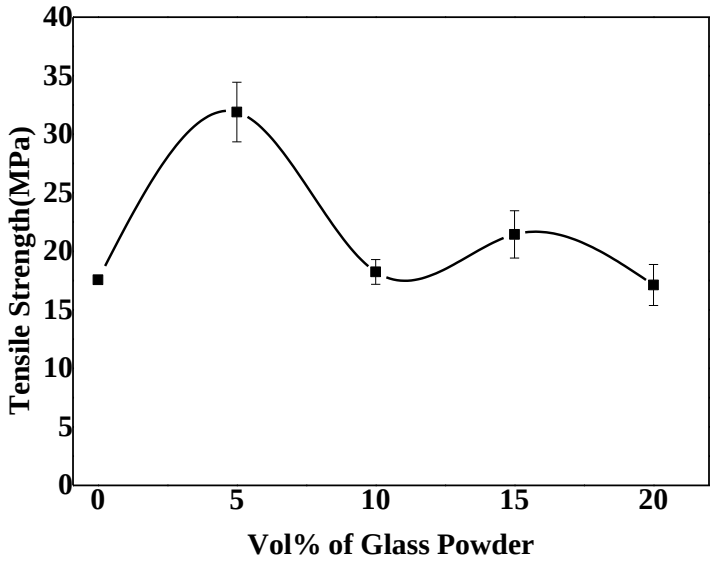


Fig-4.9 Graph shows the variation of Tensile strength vs Vol% of Glass Powder

The reason of the increase of the tensile strength of the composite upto 5 vol% is that the low volume percent filler has an advantage that it forms segregation that results a good interface bonding between matrix and filler up to a certain extent. But when we keep adding more and more fillers then the interfacial bonds are broken and hence the strength again decreases

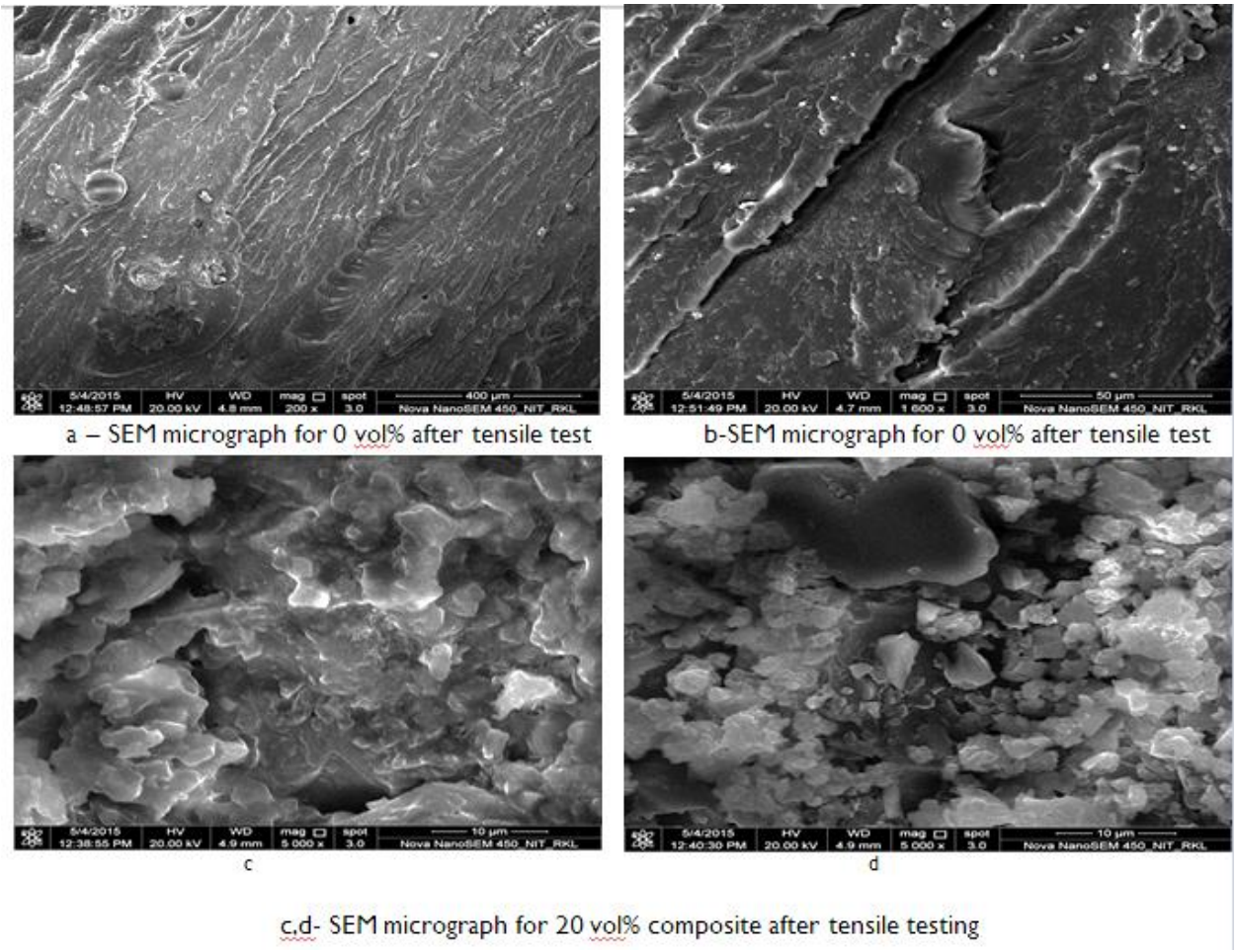


Fig-4.10: SEM images of composites after tensile testing

4.3.3 Compression Test

The compressive strength of a material is defined as the ability of the material to withstand against load when it is deformed. Also the compressive strength signifies the value for design of any structure. Generally this test is done in UTM. In this test generally 3 samples were prepared under each vol% of glass powder and tested. The results were plotted by the graphs below.

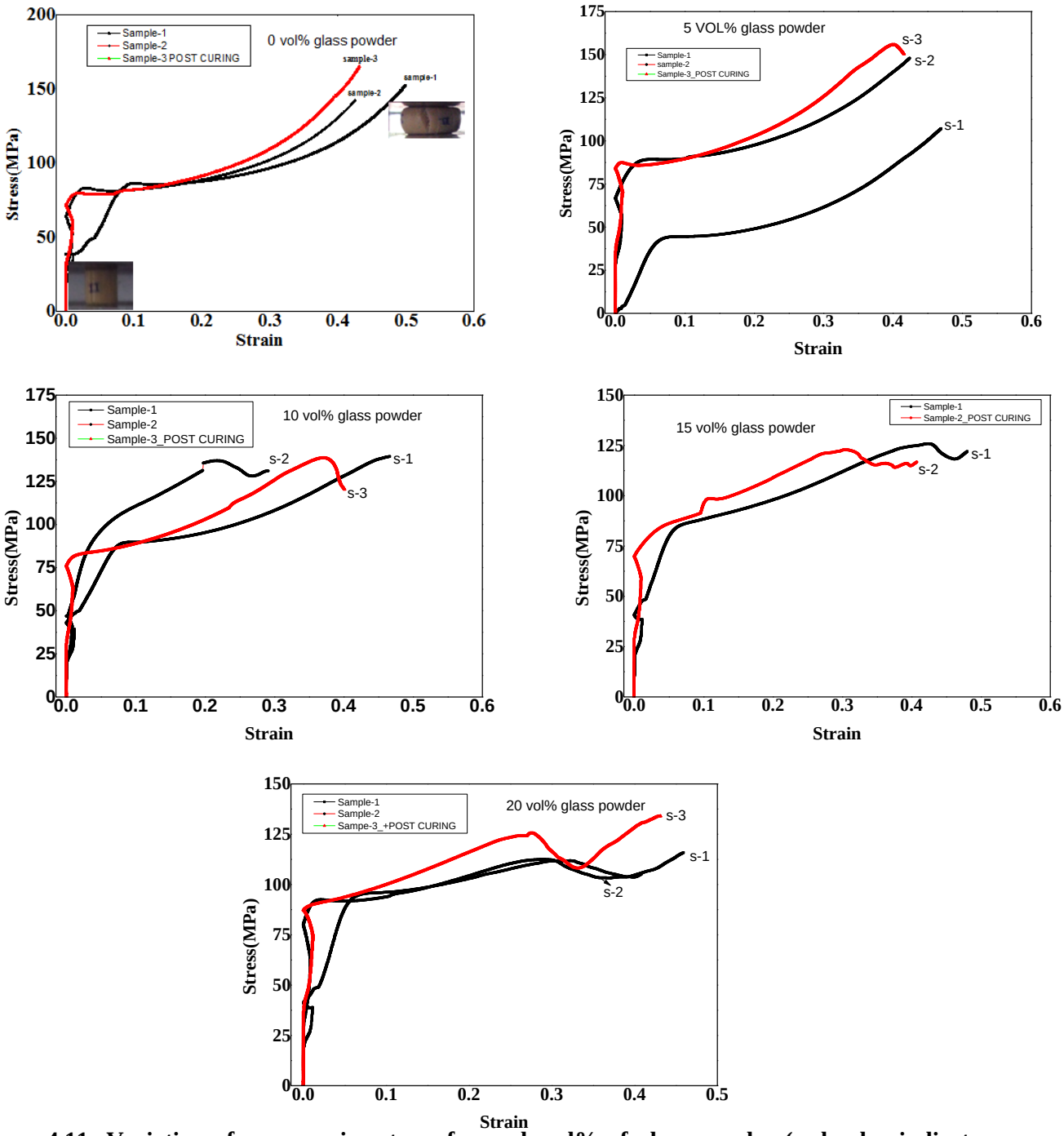


Figure-4.11: Variation of compressive stress for each vol% of glass powder (red color indicates compression for post-cured samples).

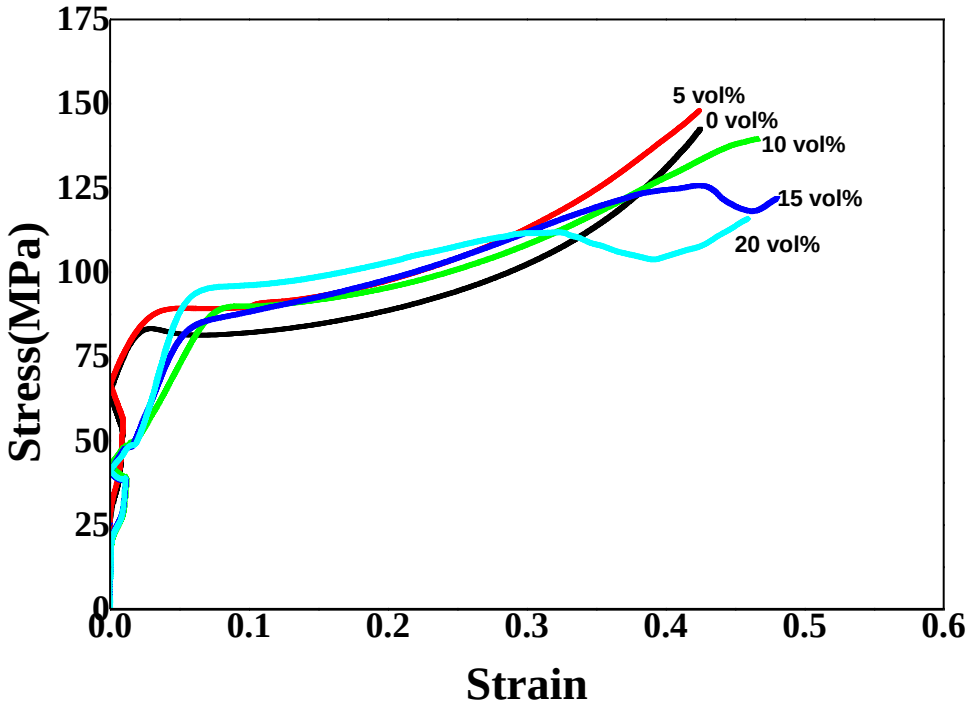


Fig. 4.12: Composite graph for Stress vs strain curves for different vol% of glass powder

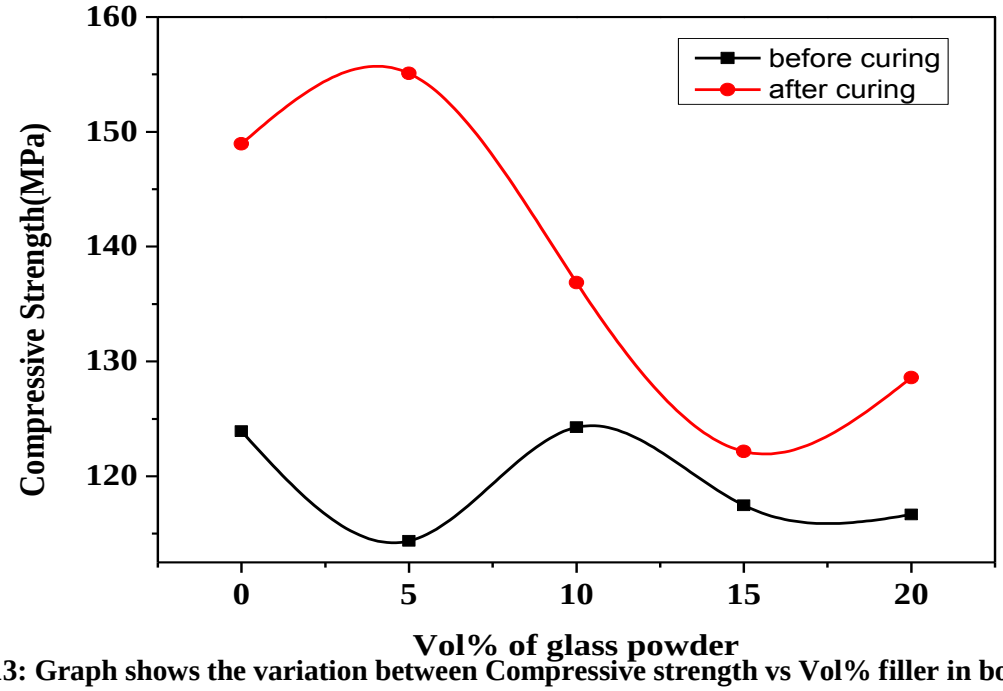


Fig-4.13: Graph shows the variation between Compressive strength vs Vol% filler in both the cases

i) before post-curing ii) after post-curing

In this case for i) before post curing the glass particulates aggregate at some places due to which the brittleness increases and hence there is the decrease of compressive stress. But for ii) after post curing the segregation of the glass powders are broken due to which the strength starts to increase up to a certain extent. Again if along with increase of the content of glass powder, brittleness increases and hence strength decreases.

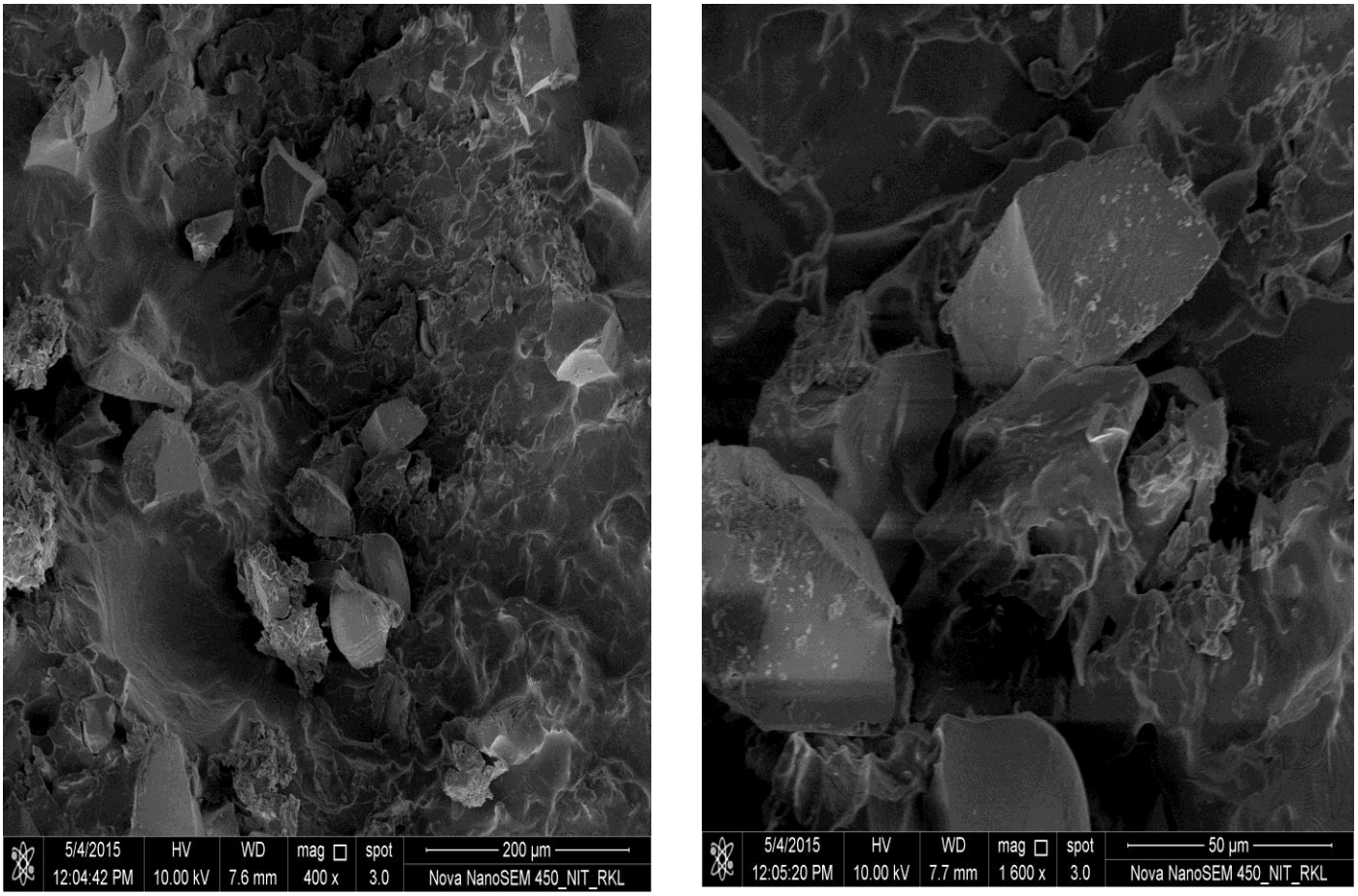


Fig-4.14 SEM micrgraphs of 20 vol% composites after compression testing

4.3.4 Impact Test

Impact is a high force applied to a body over a short range of time. In this test a hammer which is attached by a string is applied to impart the sudden force to the specimen. By this the amount of energy which is applied on the body is calculated. In this case, the Izod test has been done the sample dimension for Izod test is 63.5mm × 12.7 mm× 3.2 mm.

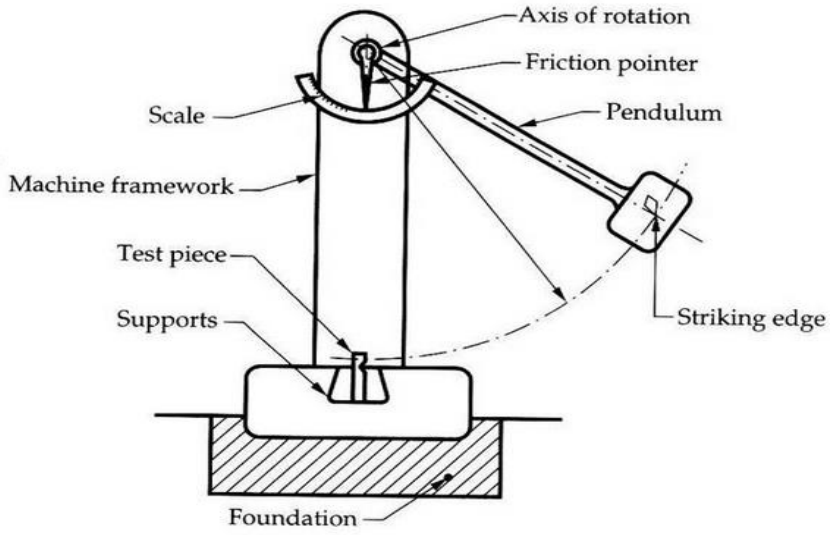


Fig- 4.15: Diagram of an Izod Impact tesing machine

fig-4.16: An Izod Testing Machine for Impact testing

The result of the impact testing has been shown in the graph below

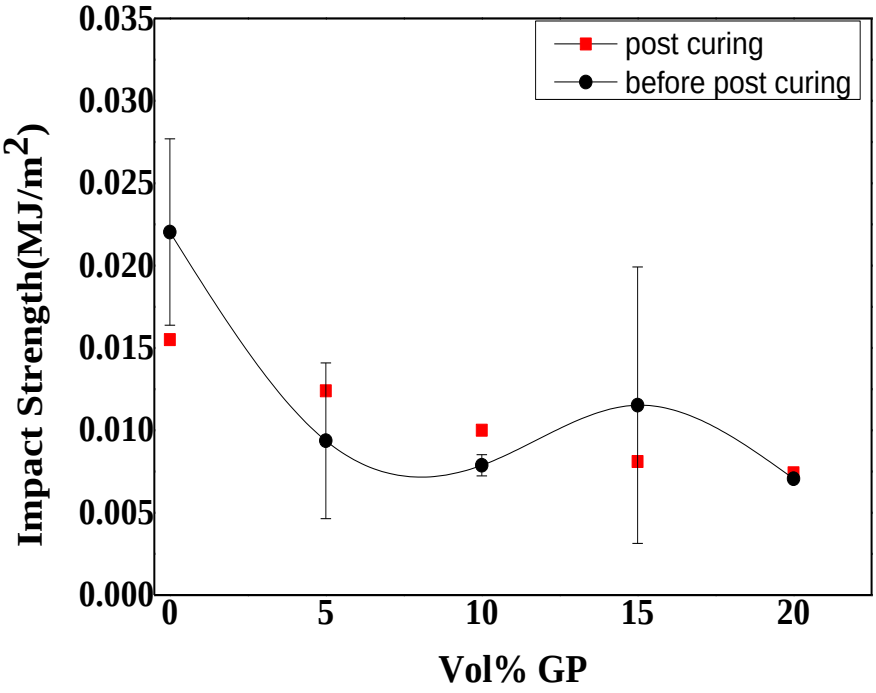


Fig- 4.17: graph between Impact Strength vs Vol% GP

For impact testing the brittleness comes to play a major role. Apart from that the addition of the glass powders more and more would increase the stress raisers in the microstructure of the composites. So the addition of glass powders decreases the impact strength.

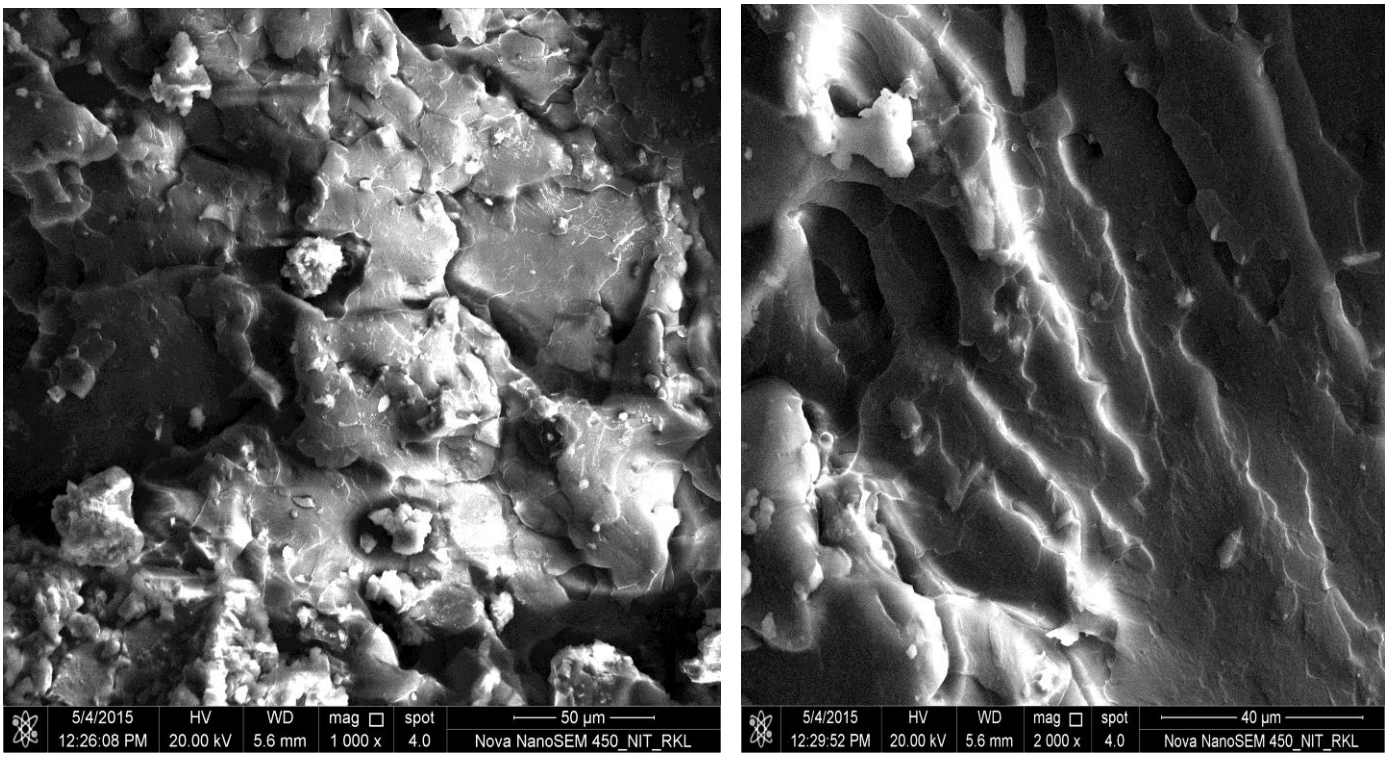


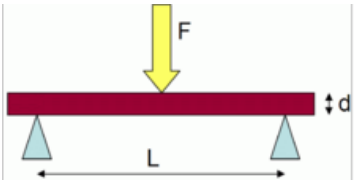
Fig-4.18 SEM micrgraphs of 20 vol% composites after impact testing

4.3.5 Flexural Test

It is also known as MOR (modulus of rupture). It is defined as the ability of a material to resist deformation when it is applied a load. It also represents the maximum stress experienced by a body at its moment of rupture. It is expressed as symbol σ.

For a rectangular sample under a load in a three-point bending setup ;

$$\sigma = \frac{3FL}{2bd^2}$$



Where

- *F* is the load (force) at the fracture point
- *L* is the span length (Here L=50mm)
- *b* is width
- *d* is thickness

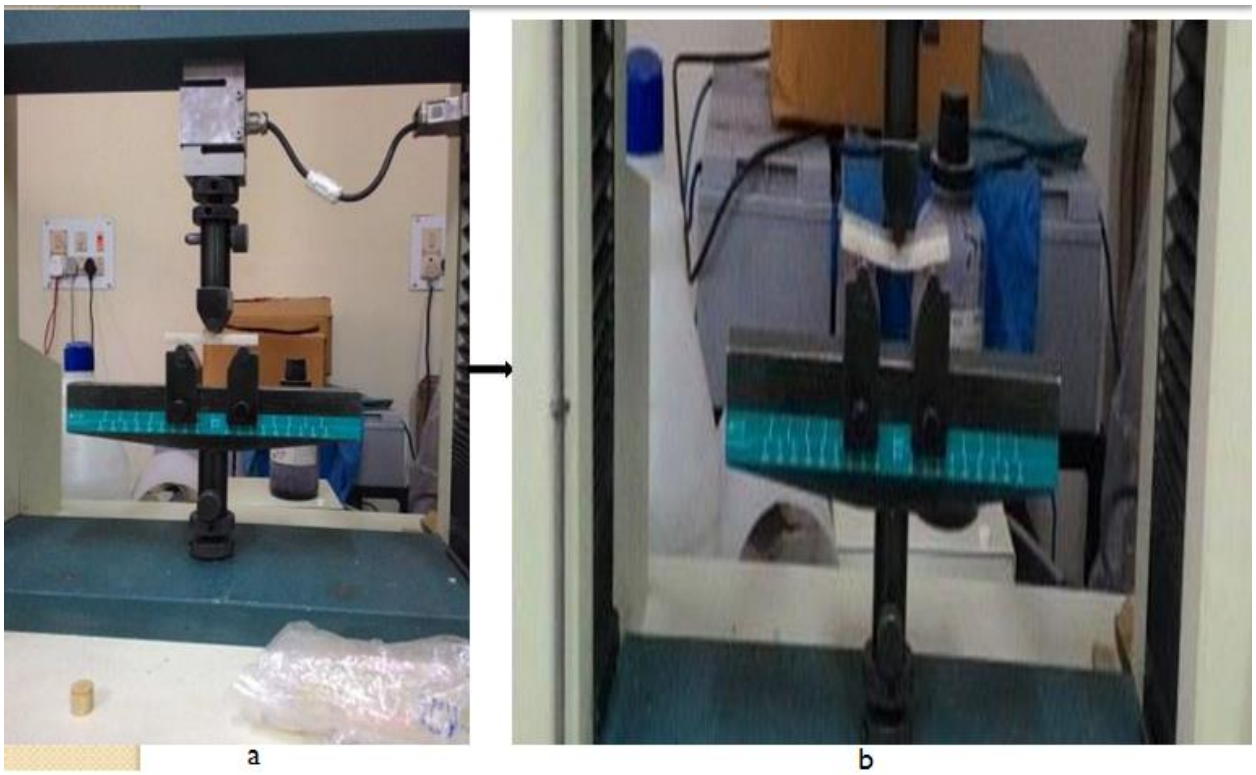


Fig-4.19: images taken when the sample (a) before testing (b) being tested meanwhile

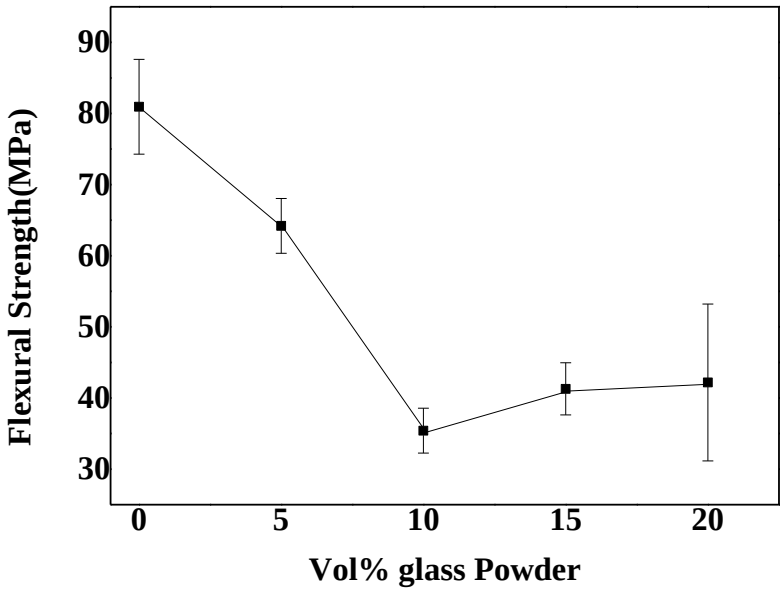


Fig-4.20: Flexural Strength vs Vol% of Glass Powder

For higher glass content fracture surface becomes rough and irregular. This change results from particle interaction with advancing of the crack. So if the increase of glass powder happens then the crack tip energy would be having more and more compared to the neat epoxy. So the flexural strength became decreased if more glass powder was added.

4.3.6 Vickers Hardness Test

This test is generally used for small parts, thin sections of a body. The principle of this test is that it uses a diamond indenter to make an indent which is measured and converted to a hardness value. A square based pyramid shaped diamond is used for testing in the Vickers scale. The HV number (Vickers Hardness number) can be determined by the following formula:

$$HV = \frac{F}{A} \approx \frac{1.8544F}{d^2},$$

where F is in kg_f and d is in millimeters.

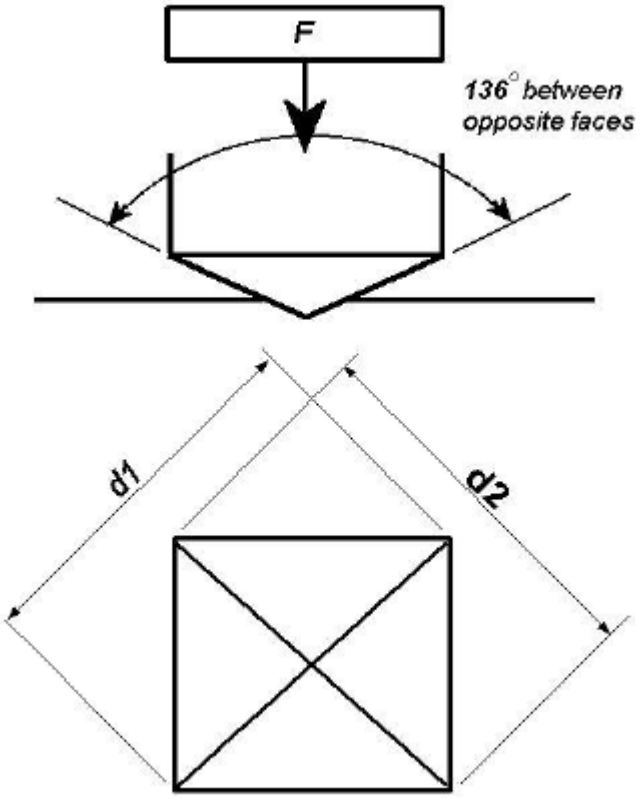


Fig-4.21: Vickers Hardness Principle

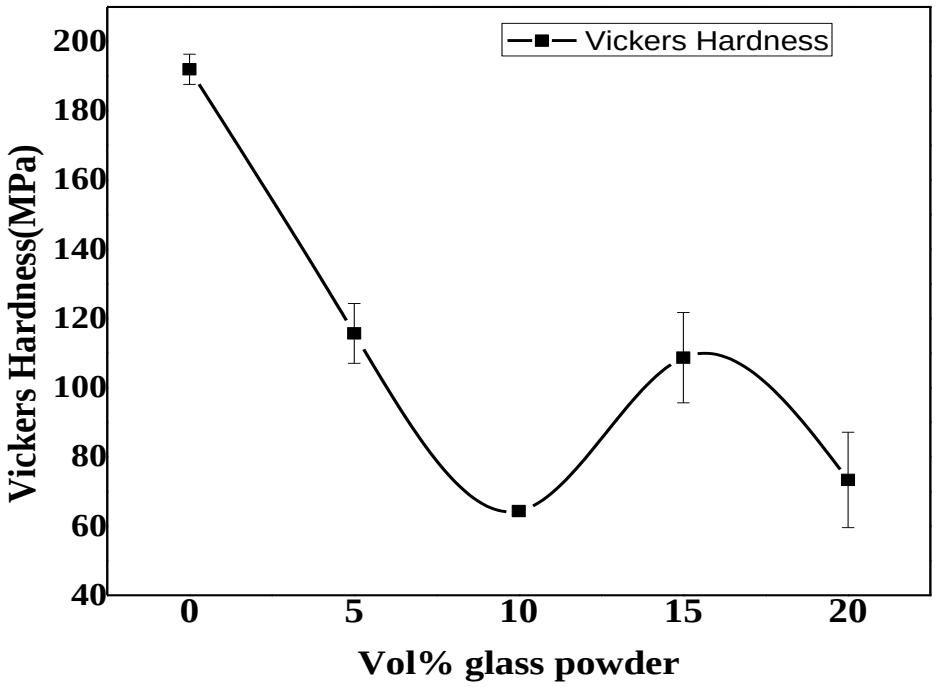


Fig-4.22: Variation of HV with Vol% of GP

The reason for decreasing in Vickers hardness with increasing the content of glass particulate is still not clear to us. Further experiments are needed in order to verify this type of trend.

Chapter 5

CONCLUSIONS

Conclusions:

From the above project following conclusions were drawn-

- The polymer matrix composite based on the waste glass material was successfully prepared.
- Tensile strength increased up to 5 vol% glass powder as reinforcement.
- Compressive strength was initially found to be decreased upto 5 vol% glass powder, but for other volume% of glass particulate, the compressive strength became comparable with 0 vol% Glass powder composite.
- Impact Strength, Flexural Strength, hardness value were decreased with the increase in the content of the reinforcement (i.e., glass particulate).
- Glass particulate was found to be effective reinforcement in case of application involving tensile and compressive load.

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